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Effect of aging type and aged unit on the repair strength of resin composite to feldspathic porcelain in testing microtensile bond strength

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Short title: *Effect of aging protocols on the repair bond strength*

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Abstract: To investigate the effect of aging type (thermocycling versus water storage) and aged unit (block versus stick) on the repair strength of resin composite to feldspathic porcelain in testing microtensile bond strength (μTBS). Ceramic specimens (N=30) (10x5.7x4.5 mm^3, Vita Mark II, Vita) were obtained from CAD-CAM blocks. One surface was etched with 10% HF and silanized. An adhesive was applied and resin composite blocks were constructed incrementally on the conditioned surface. The specimens were randomly divided into 5 groups (n=6): Control (C): Non-aged; BTC: Blocks were thermocycled (5-55°C, 6000 cycles); STC: Sticks were thermocycled; BS: Blocks aged in water storage (6 months) after themocycling; SS: Blocks aged in water storage (6 months) after thermocycling. After μTBS test, failure types were classified. Data (MPa) were statistically analyzed (1-way and Dunett and 2-way ANOVA, Tukey`s) (α=0.05). Two-parameter Weibull distribution values including the Weibull modulus, scale (*m*) and shape ($_0$), values were calculated. Aging type (p=0.009) and aged unit (p=0.000) significantly affected the results. Interaction terms were also significant (p=0.000). Considering the stick level, there was no significant difference between thermocycling (STC:25.7±2.3) and water storage (SS:25.3±3.8) (p>0.05) but the results were significantly higher when blocks were thermocycled (BTC:31.6±2.9) (p<0.05). Weibull modulus and characteristic strength was the highest in BTC (*m*=4.2; $\sigma_{o:}$34.4) among all other groups (*m*=3-3.9; $\sigma_{o:}$14.6-28.5). Adhesive failures were common and cohesive failures occurred in less than 5% in all groups. Aging protocol was detrimental on durability of repair strength of resin composite to feldspathic porcelain. Exposing the sticks to either thermocycling or water storage aging should be considered in in-vitro studies.

Keywords: Adhesion, aging, bond strength, resin composite, repair

Introduction

The introduction of adhesion to porcelains in the early 1980s with the etching ceramic surface [1] allowed the development of several ceramic materials for dental applications [2]. Improvements on the adhesion have been the focus of many studies [3-9] where two clinical scenarios were of interest, namely cementation of the restorations [5,7,9] and repair of ceramic defects using resin composites [4,8]. Essentially, in both situations resinous materials are used that are composed of organic polymer matrix and reinforced inorganic filler particles [10]. Although it is important to quantify the initial bond strength between ceramic restorations and cements or repair materials, the clinical relevance of the studies increases when the bond test is performed after aging the interface [5].

Storage of test specimens in water for periods up to 3 months, thermocycling between the extreme temperatures found in the mouth or exposing the specimens to mechanical fatigue using vertical loads are the most commons aging approaches found in the literature [11]. Storing the specimens in water for 9 months was shown to decrease the bond strength of a ceramic-resin cement-dentin assembly [5]. The mechanism of bond degradation seems be complex including the size of the adhesive area where water is indispensable for this action [12]. The water sorption decreases the frictional forces between the polymer chains of the bonding area that eventually decreases the mechanical properties of the resin [12]. Moreover, water increases the pull-out of filler particles from resin composites possibly due to the hydrolysis of the silane bond between the organic and inorganic phases of the material [13]. On the other hand, thermocycling is used in some studies [7,9] aiming to induce contraction/expansion stresses at the interface that results from the thermal expansion coefficient of the materials [12]. This can induce crack propagation along the interface and consequently debond the materials. Different thermocycling profiles have been reported, varying from 3.000 [7], 5.000 [14], 12.000 [9] to high numbers such as 40.000 cycles [15].

The effectiveness of the aging protocol can be related to the type of interface and type of test used for the individual study. In the ceramic-resin composite bond strength, the protocol of ceramic surface conditioning is detrimental for the effectiveness of the aging protocol [3]. Thus, it seems necessary to know which is the most appropriate aging protocol for the specific test method.

Microtensile bond strength (μTBS) test was introduced in dentistry by Sano et al. [16] and was proven to be more appropriate than shear tests for evaluating the bonded joints [17]. The aging process prior to the μTBS test can be performed either by exposing the bonded block (block aging) [5,7] or the sticks obtained from the bonded block (stick aging) to the aging media [9,18,19]. However, to date the literature lacks studies comparing the effectiveness of the aging methods in these two experimental situations.

The objective of this study therefore, was to investigate the effect of aging type (thermocycling versus water storage) and aged unit (block versus stick) on the repair strength of resin composite to feldspathic porcelain in testing μTBS. The null hypothesis tested was that aging effect of thermocycling or water storage would not show significant difference on the bond strength depending on the experimental unit type (block versus stick).

Materials and Methods

Specimen preparation

Feldspathic ceramic specimens (N=30) (10 mm x 5.7 mm x 4.5 mm, Vita Mark II, Vita, Bas Säckingen, Germany) were obtained from the original CAD-CAM blocks (15 mm x 12 mm x 10 mm) in a precision sectioning device (Isomet 1000, Buehler, Lake Bluff, USA). The blocks were finished with metallographic silicone carbide papers of 600 and 1000-grit in sequence and cleaned ultrasonically (Cristofoli, Sao Paulo, Brazil) in distilled water for 10 minutes.

Bonding surfaces of all ceramic blocks were etched with 10% hydrofluoric acid for 60 s and subsequently washed with air/water spray for 30 s. The blocks were ultrasonically cleaned in distilled water for 5 minutes, dried and the silane coupling agent (Rely X Ceramic Primer, 3M ESPE, Sumare, Brazil) was applied on the bonding surface during 30 s. Then, one coat of adhesive resin (2.0 Adper Single Bond, 3M ESPE) was applied and photo polymerized for 20 s (Optilux 501, Demeton, Kerr, Orange, USA). The light intensity was verified with a radiometer throughout the experiment to make sure that it was not less than 850 mW/cm^2.

The surface conditioning of the ceramic specimens was followed by the construction of the resin composite block. Resin composite (A2 TPH, Dentsply, York, USA) was applied in 2 mm increments on the bonding surface of the ceramic block until the same size with the ceramic was reached (10 mm x 5.7 mm x 4.5 mm). Each increment was photo-polymerized for 60 s. The resin composite-ceramic assemblies were stored in distilled water for 24 hours and then assigned randomly into the following 5 experimental groups (N_{block}=6 per group) according to the aging protocol:

Aging protocols

Control (C): Non-aged group. The sticks were obtained 24 h after cementation and immediately subjected to μTBS test.

BTC: Blocks were first thermocycled (MSCT-3 Plus, Erios, Sao Paulo, Brazil) (5-55°C, 6000 cycles; dwell time: 30 s) and then sticks were obtained.

STC: Initially, sticks were obtained and then subjected to thermocycling as described for BTC.

BS: Blocks were first thermocycled as described in group BTC and then stored in distilled water for 6 months at 37°C. Thereafter the sticks were obtained.

SS: Sticks were obtained and then subjected to thermocycling as described for BTC and stored in water as described in group BS.

Microtensile bond strength (μTBS) test

Each ceramic-composite block was placed in a special mold and embedded in auto-polymerizing acrylic resin (Classico, Sao Paulo, Brazil). The blocks were then sectioned into sticks in a precision sectioning saw (Isomet 1000) under low speed (250 rpm) and water-cooling. After the first cut, the blocks were rotated 90° for the second cut aiming to obtain sticks measuring approximately 1 mm thickness. The sticks from the outer sections were discarded. Each resultant stick had bonding area measuring 1±0.1 mm^2 and length of approximately 9 mm. From each block 14 sticks were obtained.

Bonding area of each stick was measured with a digital caliper (Starret Industria e Comercio Ltd, Itu, Brazil) again and noted prior to tests. Each stick was then fixed in the jig using cyanoacrylate gel (Super Bonder Gel, Loctite Ltd, Sao Paulo, Brazil) parallel to the long axis of the device, which was fixed in a universal testing machine (DL-1000, EMIC, São José dos Pinhais, Brazil). The sticks were loaded in tensile at a crosshead speed of 1 mm/min with a 10 Kgf load cell, until the failure. 10 Kgf was the maximum load that the load cell could support. This load amounts approximately 98 N, which was more than sufficient for the present study where the highest values were about 50 N.

The maximum load to failure was recorded using the software programme (TESC, EMIC, São José dos Pinhais, Brazil). The μTBS was calculated according to the equation R = F/A, where R was the strength (MPa), F was the load (N) required to debond the specimen, and A was the bonding area (mm^2).

Failure analysis and microscopy evaluation

Failure sites were initially observed using an optical microscope (Zeiss Stemi 2000-C, Edmund Optics Inc., Barrington, USA) at x50 magnification and classified as follows: Type I: Adhesive failure between the ceramic and the resin composite; Type II: Cohesive failure in the ceramic or the resin composite; Type III: Mixed failure between the ceramic and the resin composite with more than half of the composite remained on the ceramic surface.

Additionally, representative sticks from each group were selected to complementary analysis under scanning electron microscopy (SEM, Jeol-JSM-T330A-Scanning Microscope, Tokyo, Japan) at 15 kV using secondary electron mode at a magnification of x220.

Statistical analysis

A sample size of 6 blocks in each group was calculated to have more than 80% power to detect a difference of 5 MPa between mean values (Satterthwaite t-test (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL, USA) with a 0.05 two-sided significance level,.

Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data (Minitab Software V.16, Minitab Inc., Centre County, USA). As the data (MPa) were normally distributed, 1-way analysis of variance (ANOVA) and Dunett and 2-way ANOVA and Tukey`s post-hoc tests were applied at two levels (Level 1: Thermocyling versus water storage; Level 2: Block versus stick) to analyze the possible significant differences between the groups. The blocks were considered as experimental units. Maximum likelihood estimation without a correction factor was used for 2-parameter Weibull distribution (Weibull^{++}, ReliaSoft, Tucson, USA), including the Weibull modulus, scale (*m*) and characteristic strength (σ_o) in order to interpret predictability and reliability of adhesion considering the sticks as the experimental unit.

Results

No pre-test failures were experienced during obtaining sticks or aging process.

Aging type (p=0.009) and aged unit (p=0.000) significantly affected the results. Interaction terms were also significant (p=0.000) (Table 1).

Considering the stick level, there was no significant difference between thermocycling (STC:25.7±2.3) and water storage (SS:25.3±3.8) ($p>0.05$) but the results were significantly higher when blocks were thermocycled (BTC:31.6±2.9) ($p<0.05$) (Fig. 1, Table 2).

Weibull modulus and characteristic strength was the highest in BTC (m=4.2; $\sigma_{o:}$34.4) among all other groups (m=3-3.9; $\sigma_{o:}$14.6-28.5) (Fig. 2, Table 1).

Adhesive failures were common and cohesive failures occurred in less than 5% in all groups (Table 3, Figs. 3a-b).

Discussion

Aging the bonded interfaces prior to adhesion tests is an essential procedure that gives information on long-term clinical durability of the adhesive dental applications. For this reason, this study was undertaken to investigate the effect of aging type (thermocycling versus water storage) and aged unit (block versus stick) on the repair strength of resin composite to feldspathic porcelain. Based on the results of this study, since both aging method and the aged unit significantly affected the results, the null hypothesis cold be rejected.

Storage in water for a period of 3 months results in water sorption, while thermocycling aims to induce some kind of contraction and expansion stresses at the bonded interface [12]. Both aging methods are expected to reduce the bond strength of the system [4,5]. However, the effectiveness of the aging procedure depends on some factors, such as materials bonded ceramic-composite, ceramic-cement, reinforced ceramic-resin composite, reinforced ceramic-cement, ceramic-resin cement-dentin assemblies [5,8,19] or surface conditioning methods (i.e. etching, air-borne particle abrasion) [6,19]. In addition to these factors, bonding test type (tensile, microtensile, shear, microshear) [20] and tests design (i.e. hourglass, dumbbell or stick shaped specimen for the μTBS test) [21] all affect the bond strength results. Thus, comparison between the available studies in the literature, should be made in caution.

In the present study, the aging process did not affect the bond strength of the BS group in which the blocks were aged in water for 6 months compared to the control group (C). It has to be also noted that these groups delivered the lowest bond strength and the highest number of adhesive failures. Conversely, when blocks were thermocyled (BTC), significantly higher results were obtained. This could be explained on the grounds that thermocycling especially exposure to 55°C bath, probably further contributed to polymerization of the bonded joint. In another study, testing the bond strength between zirconia and resin cement where porcelain-resin cement was considered as the control group [19], also reported increased bond strength after sticks were stored in water for 1 and 3 months. However, after 6 months the results were similar to the non-aged group. It is possible that the resin present at the interface takes several months to complete the polymerization and becomes stable, which can explain the higher results of SS group compared to control. Moreover, contact with hot water can accelerate this process. Contrary to this study, Guarda et al. [4] found reduction in the μTBS between ceramic and resin cement after thermocycling the block (5-55°C). Yet, the number of cycles was limited to 3.000 cycles. Another study, employed thermcycling on sticks (5-55°C; 12.000) with additional storage at 37°C for 150 days and did not find significant differences between aged and non-aged specimens [7].

When blocks are aged, technically the marginal sticks obtained from the blocks are discarded in μTBS tests. This is due to possible excess cement or deficiencies at the margins [22]. In fact, only margins of the block are directly in contact with water during the whole course of storage time. Therefore, only the sticks obtained from the internal part of the blocks are used for μTBS test. The non-significant bond strength between the STC and SS in this study indicates similar type of aging due to limited bonding area of $1mm^2$. Similar Weibull moduli (STC: 3.4; SS: 3.5) in these two groups also support this assumption.

Nevertheless, it has to be emphasized that the discrepancy between the studies, could be highly related to the number of cycles during thermocycling, possible further polymerization or expansion of the resin material that may yield to even increase in bond strength after aging conditions. Further studies should identify critical aging conditions that could identify severe aging affect on bonded joints of ceramic-resin interface.

Conclusions

From this study, the following could be concluded:

1. Aging protocol and aged unit significantly affected the durability of repair strength of resin composite to feldspathic porcelain when μTBS test was used.
2. Exposing the sticks instead of ceramic-resin composite blocks to either thermocycling or 6 months water storage aging should be considered in in-vitro studies to achieve more effective aging of the bonded interfaces.
3. Failure types were predominantly adhesive failure type between the ceramic and resin composite followed by mixed failures.

Clinical Relevance

In vitro studies on repair of feldspathic porcelain with resin composite should be evaluated with caution. Only those that practiced aging on bonded sticks should be considered as more worse-case scenario for long-term clinical durability of repairs.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to tables and figures:

Tables:

Table 1. Microtensile (μTBS) bond strengths (Mean ± standard deviation) of resin composite to feldspathic porcelain after aging methods applied on either blocks or sticks, Weibull modulus (*m*) and characteristic strength (σ_o). Same lower-case letters in each column indicate no significant differences (α=0.05). C: Control group; BTC: Blocks thermocycled; STC: Sticks thermocycled; BS: Blocks stored in water for 6 months; SS: Sticks stored in water for 6 months.

Table 2. Microtensile (μTBS) bond strengths (Mean ± standard deviation) of resin composite to feldspathic porcelain as a function of aging method and aged unit. Same lower-case letters in each column indicate no significant differences (2-way ANOVA, Tukey`s, α=0.05). See Table 1 for group abbreviations.

Table 3. Frequencies of failure modes in percentages. Type I: Adhesive failure between the ceramic and the resin composite; Type II: Cohesive failure in the ceramic or the resin composite; Type III: Mixed failure between the ceramic and the resin composite with more than half of the composite remained on the ceramic surface.

Figures:

Fig. 1 Microtensile (μTBS) bond strengths (Mean ± standard deviation) of resin composite to feldspathic porcelain after aging methods (Thermocycling-TC; Water storage for 6 months-S) applied on either blocks (B) or sticks (S).

Fig. 2 Probability plot with Weibull curves (95% CI) using maximum likelihood estimation. The 95% confidence intervals of *m* parameter overlapping indicate no statistical difference.

Note that BTC presented higher σ_o and that there was no significant difference between the σ_o values of SS and STC and C and BS.

Figs. 3a-b SEM images (x220) of **a)** Stick with adhesive failure. The right larger image shows the overview of the bonding surface of the ceramic part. The left smaller image shows the lateral view of the bonding area of the tested stick in stereomicroscope, including the ceramic and the resin parts. **b)** Stick with mixed failure. The right larger image shows the overview of the bonding surface of the ceramic part. The white arrows indicate the regions where the failure becomes cohesive of in the ceramic (A). The left smaller image shows the lateral view of the bonding area of the tested stick in stereomicroscope, including the ceramic and the resin parts.

Tables:

Experimental Groups	N_{block}	Mean (SD)	*m*	σ_o
C	6	14.4 (1.3)[a]	3.9	15.8
BTC	6	31.6 (2.9)[b]	4.2	34.4
STC	6	25.7 (2.3)[c]	3.4	28.5
BS	6	13.1 (0.6)[a]	3	14.6
SS	6	25.3 (3.8)[c]	3.5	28

Table 1. Microtensile (μTBS) bond strengths (Mean ± standard deviation) of resin composite to feldspathic porcelain after aging methods applied on either blocks or sticks, Weibull modulus (*m*) and characteristic strength (σ_o). Same lower-case letters in each column indicate no significant differences (α=0.05). C: Control group; BTC: Blocks thermocycled; STC: Sticks thermocycled; BS: Blocks stored in water for 6 months; SS: Sticks stored in water for 6 months.

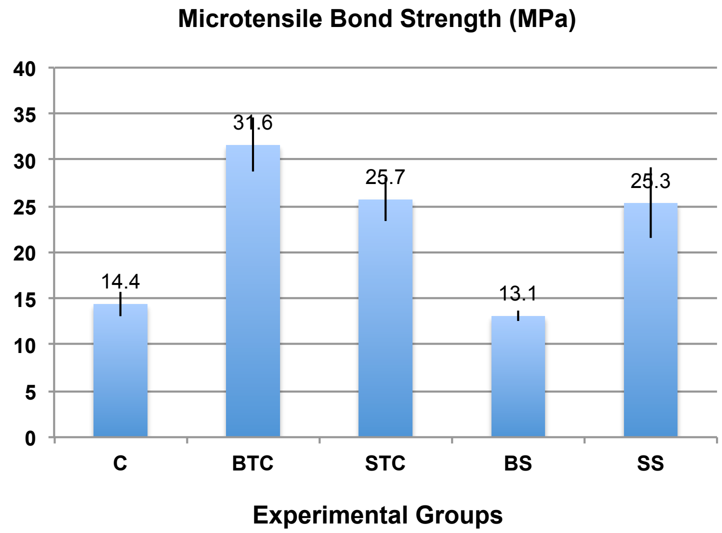
Aging Method	Unit	N_{block}, N_{stick}	Mean (SD)
Thermocycling	Block	6, 14	31.6 (2.9)[a]
	Stick	6, 14	25.7 (2.3)[b]
Water storage	Block	6, 14	13.1 (0.6)[c]
	Stick	6, 14	25.3 (3.8)[b]

Table 2. Microtensile (μTBS) bond strengths (Mean ± standard deviation) of resin composite to feldspathic porcelain as a function of aging method and aged unit. Same lower-case letters in each column indicate no significant differences (2-way ANOVA, Tukey`s, α=0.05). See Table 1 for group abbreviations.

Experimental Groups	N_{block}	N_{stick} (%)	Type I (%)	Type II (%)	Type III (%)
C	6	84 (100)	52 (61.9)	4 (4.8)	28 (33.3)
BTC	6	78 (100)	40 (51.3)	3 (3.8)	35 (44.5)
STC	6	82 (100)	44 (53.7)	4 (4.9)	34 (41.5)
BS	6	86 (100)	53 (61.6)	3 (3.5)	30 (34.9)
SS	6	83 (100)	50 (60.2)	2 (2.4)	31 (37.3)

Table 3. Frequencies of failure modes in percentages. Type I: Adhesive failure between the ceramic and the resin composite; Type II: Cohesive failure in the ceramic or the resin composite; Type III: Mixed failure between the ceramic and the resin composite with more than half of the composite remained on the ceramic surface.

Figures:



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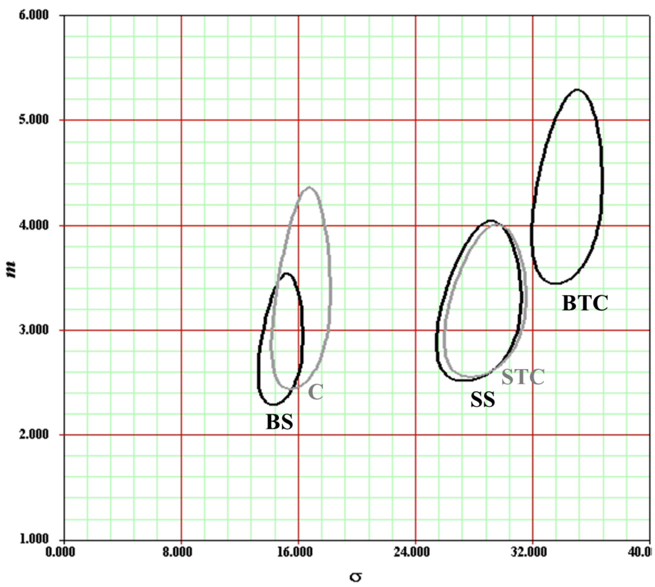

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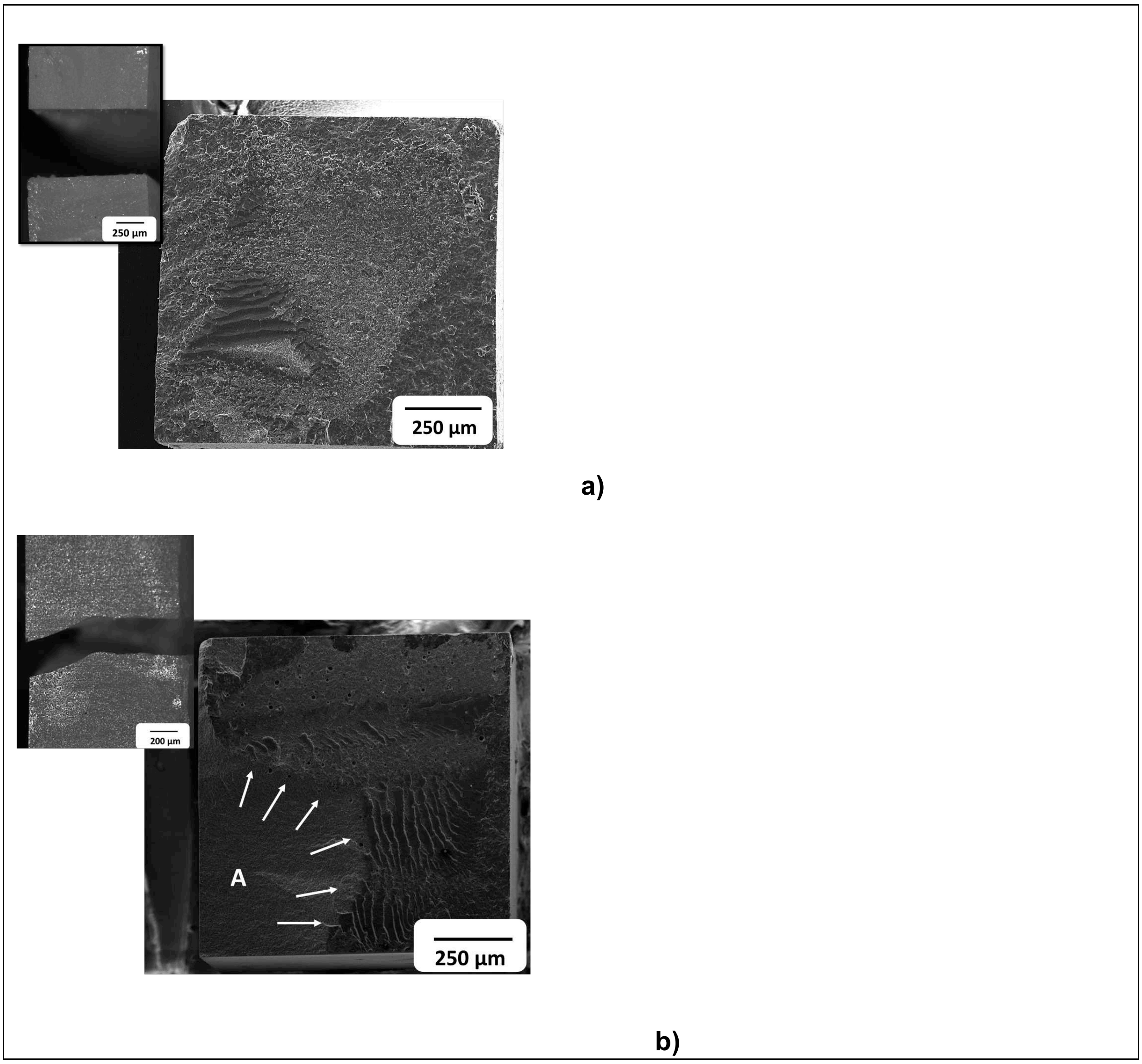

Figs. 3a-b SEM images (x220) of **a)** Stick with adhesive failure. The right larger image shows the overview of the bonding surface of the ceramic part. The left smaller image shows the lateral view of the bonding area of the tested stick in stereomicroscope, including the ceramic and the resin parts. **b)** Stick with mixed failure. The right larger image shows the overview of the bonding surface of the ceramic part. The white arrows indicate the regions where the failure becomes cohesive of in the ceramic (A). The left smaller image shows the lateral view of the bonding area of the tested stick in stereomicroscope, including the ceramic and the resin parts.